IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re Application of:	Donald R. Huffman *et al.*	Confirmation No.:	9010
Serial No.:	08/471,890	Group Art Unit:	1795
Filed:	June 7, 1995	Examiner:	YUAN, Dah-Wei

FOR: NEW FORM OF CARBON

Technology Center Director Jacqueline Stone
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

PETITION TO TECHNOLOGY CENTER DIRECTOR

OF GROUP ART UNIT 1795

This petition is submitted to the Technology Center Director of Group Art Unit 1795 pursuant to 37 CFR §1.181 and MPEP §1002.02(c)(3)(h), in response to the Examiner's decision in the final Office Action of June 23, 2010, to not enter Applicants' Supplemental Amendment filed September 25, 2009. Applicants respectfully request the Director's intervention as to the Examiner's decision not to enter the Supplemental Amendment, for the reasons detailed below. This petition is timely filed within two months of the date of the final Action as set forth in 37 CFR §1.181. A Request for Reconsideration containing these arguments was filed with the Examiner on July 16, 2010, but as of this date, no further Action has occurred.

Background

This application has had a long prosecution history marked by many retracted indications of allowability as detailed in section 3 of the legal arguments below. This application has a priority date back to 1990, was filed pre-GATT on June 7, 1995, and is still pending over fifteen years later. In the final Office Action dated June 23, 2010, the Examiner sets forth a new rejection under 35 U.S.C. §112, first paragraph, never raised before on the record. For that reason alone, the finality of the Office Action should be withdrawn and Applicants' Supplemental Amendment should be entered, as detailed in section 2 of the legal arguments below. Indeed, this new rejection clearly was not made as a result of Applicants' amendment and response to the non-final Office Action, but rather as a result of yet another change in examiner and a difference of opinion between the multiple examiners that have been assigned to this case over the years.

This new rejection under §112, first paragraph was first raised orally by Examiner Yuan after Applicants submitted their response to the non-final Action on March 12, 2009. Applicants have tried in good faith to respond to Examiner Yuan's concerns regarding claim scope by proposing responsive claim amendments as detailed in section 1 of the arguments below. Instead of giving those proposed amendments due consideration, Examiner Yuan has essentially required that Applicants accept only the language in Examiner Yuan's amendment, and yet has provided Applicants with no valid reasons as to why their proposed claim amendments would not be sufficient. Likewise, Examiner Yuan now refuses to enter Applicants' Supplemental Amendment filed September 25, 2009, despite the fact that the Supplemental Amendment contains proposed claim amendments that respond directly to this newly raised ground of rejection. Moreover, because the Action dated June 23, 2010 has been made final and amendments after final are only entered at the discretion of the Examiner, Applicants presume that Examiner Yuan will also refuse

to enter Applicants' amendments should they be submitted in response to the final Action. Consequently, only an RCE, and loss of pre-GATT status (leading to expiration of this application), would facilitate entry of Applicants' proposed amendments.

The cumulative effect of these actions is that Applicants have been robbed of their right to a complete examination on the merits with regard to the newly raised §112, first paragraph rejection. The Office has essentially raised a new ground of rejection that was not justified by Applicants' amendment and has made the rejection final – indeed, the claim language that Applicants employed in the amendment submitted March 12, 2009 was previously found to be equivalent to the language proposed by Examiner Yuan and therefore allowable. By making this rejection now, after final, and cutting off Applicants' right to make amendments in response to newly raised rejection, the Office has in effect acted in an arbitrary and capricious manner and has denied Applicants the requisite due process as set forth in the rules.

Legal Basis for Entry of Supplemental Amendment

1. **Contrary to the Examiner's position, the claims in the Supplemental Amendment do adopt suggestions made by the Office and also limit the issues for appeal, and entry is therefore appropriate pursuant to 37 CFR 1.111(B) and (F)**

According to 37 CFR 1.111(a)(2) (the provision relied upon by the Examiner), a Supplemental Reply may be entered if the Supplemental Reply is limited to (B) adoption of the examiner suggestions(s) and (F) simplification of the issues for appeal. As the Examiner has already acknowledged, claims 164-201 are duplicates of those suggested by Examiner Yuan himself and therefore clearly fall under provision (B) as adopting the Examiner's suggestions. Thus, it appears to be claims 202-245 that are the Examiner's primary reason for not entering the

amendment. Claims 202-245, however, also incorporate amendments that were suggested by the Office and also address concerns raised by Examiner Yuan himself, and in any case clearly simplify the issues for appeal under section (F) of the rule.

For example, as acknowledged by Examiner Yuan in the final Office Action, Examiner Hendrickson previously indicated that claims containing language limiting the "cage" to 60 or 70 carbon atoms would overcome a putative rejection under 35 U.S.C. §112 and would be allowable. *See* final Office Action, page 5. Applicants are agreeable to this suggestion and have incorporated the suggested language into the Supplemental Amendment. *See* newly proposed claims 203, 204, 216, 217, 224, 225, 236, and 237. Because the new claims incorporate a suggestion made directly by the Office, Applicants believe that the amendment meets the requirements of 37 CFR 1.111(a)(2).

Furthermore, the newly proposed claims are limited to compositions comprising fullerenes having a polyhedral, spherical or icosahedral structure. *See* newly proposed claims 202, 209-211, 215, 221, 222 and 239-245. As Applicants explained to Examiner Yuan during the telephone conference of September 24, 2009, Applicants believe that inclusion of the terms "polyhedral," "spherical" and "icosahedral" in these claims most clearly responds to the Examiner's concern that the claims not be so broad as to read on carbon nanotubes (which contain open ended cylindrical structures). This concern is now reflected in the Examiner's rejection of the previously pending claims under 35 U.S.C. §112, first paragraph, where the Examiner has rejected the pending claims as being broad enough to read on carbon nanotubes. (*see* page 11 of Office Action). Yet, the Examiner refuses to enter Applicants' proposed claims that would clearly address this concern, instead forcing Applicants to accept the Examiner's

proposed claim language, which Applicants believe is confusing and furthermore, does not adequately address the concern that the Examiner himself raised.

For example, the Examiner has proposed to limit all claims to read on "cage molecules consisting of carbon atoms that are soluble in non-polar solvents." *See* proposed Examiner's Amendment. The Examiner's rationale for this proposed amendment is that limiting the cage molecules to those that are soluble in non-polar solvents will exclude carbon nanotubes. *See* Office Action at pages 6 and 11.[1] However, the Examiner's proposed amendment does not effectively accomplish the intended exclusion, as solubility in nonpolar solvents is a variable attribute that may be altered by the inclusion of other chemical entities.[2] Moreover, the Examiner's proposed claims arguably suffer from §112, second paragraph issues in that it is unclear as to whether it is the cage molecule or the carbon atoms themselves that are soluble in non-polar solvents.

The Examiner further argues on page 6 of the Office Action that the term "fullerene" was never discussed in any interview prior to September 2009 nor addressed in any of the previous Office Actions of record. Applicants respectfully disagree. It is the Office itself that began using the term "fullerene" in the first non-final Office Action dated June 3, 1996. Furthermore, in the non-final Office Action dated September 12, 2008, the prior Examiner noted that the terms "fullerene," "a cage carbon allotrope consisting solely of carbon atoms soluble in non-polar organic solvents, which allotrope is neither graphite or diamond," and "a cage carbon consisting

[1] The Examiner clearly ignores the structural limitations in Applicant's proposed claims in the Supplemental Amendment, again asserting that "fullerenes" read on carbon nanotubes (see page 6 of Office Action).
[2] For example, Ji et al. report the use of polysiloxane surfactants to render carbon nanotubes soluble in nonpolar solvents. Ji et al., 2009, "Polysiloxane Surfactants for the Dispersion of Carbon Nanotubes in Nonpolar Organic Solvents," *Langmuir*, 2009, *25* (20), pp 12325–12331.

solely of carbon atoms" all mean the same thing.[3] Therefore, Applicants do not agree that the term "fullerene" has been newly introduced into prosecution. Moreover, the prior Examiner equated the term fullerene with the phrase Examiner Yuan is now suggesting in the proposed Examiner's Amendment. The use of the term "fullerene" in Applicants' proposed claims is therefore not a new development to the prosecution of this case.[4]

Examiner Yuan asserts in the final Office Action that "[b]y not accepting the examiner's amendments, it appears that applicants consider the limitations 'cage molecules consisting of carbon atoms that are soluble in non-polar solvents' and 'cage molecules consisting of carbon atoms' to be different in scope and not synonymous." *See* paragraph bridging pages 6-7 of final Office Action. Applicants do not understand the Examiner's point here since none of the newly submitted claims are directed solely to "cage molecules consisting of carbon atoms." In fact, in every instance in the newly submitted claims Applicants have incorporated structural limitations to distinguish the claimed compounds from carbon nanotubes, which was the main issue that Examiner Yuan raised in the telephonic interviews. Applicants simply believe that the language proposed by Applicants is a better solution to the issue raised by Examiner Yuan than the language that the Office has proposed.

Given that newly submitted claims 202-245 in the Supplemental Amendment incorporate the amendments as suggested by Examiner Hendrickson, respond directly to the concerns raised by Examiner Yuan in the multiple conferences with the Applicant, and actually address the newly posed §112 rejection more clearly and directly than the current proposed Examiner's

[3] Claims 89-92 (now cancelled), for example, used the term "fullerene."
[4] The final Office Action dated June 23, 2010 also asserts that the term "fullerene" was not used in the original disclosure (p. 6). US case law makes it clear that exact literal support in the specification is not required. *See*

Amendment and therefore certainly limit the issues for appeal, it is entirely appropriate for the Office to enter the Supplemental Amendment pursuant to 37 CFR 1.111(B) and (F).

2. Entry of the Supplemental Amendment is warranted pursuant to 37 CFR §1.112, or alternatively, the Office Action should be made non-final

According to 37 CFR §1.112, after reply by applicant to a non-final action, the application will be reconsidered and again examined. The applicant will be notified if claims are rejected, objections or requirements made, or decisions favorable to patentability are made, in the same manner as after the first examination. Applicant may then reply to such Office action in the same manner provided in §1.111, *with or without amendment*, unless such Office action indicates that it is made final or an appeal has been taken.

As summarized in the final Office Action, Examiner Hendrickson first raised an issue under 35 U.S.C. §112 in a telephonic interview with Applicants on March 12, 2009, prior to Applicants' submission of the Amendment on that same date. As Examiner Yuan acknowledges, Examiner Hendrickson "indicated that he would soon issue a rejection under §112 in a new Office Action on the ground that the term 'cage' other than 60 carbon atoms or 70 carbon atoms are not enabled by the application as originally filed." *See* page 5 of final Office Action. Thus, Examiner Hendrickson acknowledged on March 19, 2009 that there was an alleged §112 rejection that he believed should have been raised in the initial non-final Office Action that was never raised.

Examiner Yuan has continued asserting alleged §112 issues in multiple conferences since taking over the case, particularly over the issue as to whether the claims read on carbon

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Martin v. Johnson, 454 F.2d 746, 751, 172 USPQ 391, 395 (CCPA 1972) (stating "the description need not be in

nanotubes. Applicants believe that these communications by Examiners Hendrickson and Yuan constitute further examination under 37 CFR §1.112 and further action by the Office prior to any action being made final. Applicants have the right pursuant to 37 CFR §1.112 to respond to such further non-final rejections "with or without amendment."

In any case, as noted above, the non-final Office Action dated September 12, 2008 did not include any rejection under 35 U.S.C. §112, first paragraph. In fact, the Action dated September 12, 2008 indicated that some claims were allowable.[5] It appears that the §112 rejection that is now being raised in this final Office Action is a clear departure from the Office Action dated September 12, 2008. Indeed, the claims pending at the time of the non-final Office Action did not contain either the limitation proposed by Examiner Hendrickson or the limitations now proposed by Examiner Yuan and were deemed, in part, allowable. Accordingly, Applicants' amendment on March 12, 2009 did not necessitate this new ground of rejection, as asserted in the final Office Action. To be complete, the §112 issue should have been first raised in the non-final Office Action dated September 12, 2008, to allow Applicant sufficient opportunity, on the record, to address the rejection. Instead, the Office has attempted on multiple occasions to force Applicants to narrow their claims after the non-final Office Action was mailed, to avoid alleged §112 issues that were never properly raised and that Applicant has never had the opportunity to respond to on the record. In light of these facts, Applicants submit that the finality of the Office Action dated June 23, 2010 should be withdrawn.

In summary, even if the Office should deny entry of the Supplemental Amendment under 37 CFR §1.111 as not limiting the issues for appeal or responding to the Examiner's

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ipsis verbis [i.e., "in the same words"] to be sufficient").

requirements, Applicants believe that they have a right to entry of the amendment pursuant to 37 CFR §1.112 given that the issues under 35 U.S.C. §112 raised by the Examiners after the nonfinal Office Action constitute additional examination prior to final to which Applicants have a right to respond with amendments. Alternatively, the finality of the most recent Office Action dated June 23, 2010, should be withdrawn.

3. Equity warrants entry of the Supplemental Amendment

In addition to the legal provisions provided above, Applicants respectfully submit that entry of the Supplemental Amendment filed September 25, 2009 is warranted on equitable grounds. Applicants believe that while the Examiner has generally cited select portions of the prosecution correctly, he has failed to properly document the inconsistent and confusing behavior on the part of the Office and has minimized the role the Office has played in extending the prosecution of this application. As detailed below, the convoluted approach taken by the Office has essentially robbed Applicants of their right to a full and proper prosecution on the merits, and equity warrants that Applicants' Supplemental Amendment now be entered and considered on the record.

Applicants first note that on December 12, 2006, Examiner Hendrickson issued a non-final Office Action, rejecting claims 89-93 and 95-121 under 35 U.S.C. §§ 102, 103, and/or 112, and specifically noted that the specification did not support the term "macroscopic." *See* page 3 of non-final Office Action, dated December 14, 2005.

[5] For example, claim 120 was indicated as being allowable, and was directed to "Substantially pure fullerenes in solid form."

Nearly six months later, on June 8, 2007, Examiners Tsang-Foster and Chaney withdrew Examiner Hendrickson's non-final Office Action and issued a suspension of prosecution in the application as a result of a district court ruling on the sister case, U.S. Appl. No. 08/236,933. *See* Office Communication, dated June 8, 2007. Applicants were given three months to provide additional support for the patentability of the applications, specifically regarding support for the term "macroscopic." *Id.* at 3. Applicants responded on September 7, 2007 and provided the executed declarations of Drs. Terrones, Darwish and Kroto. *See* Communication, dated September 4, 2007, received September 7, 2007. Applicants provided over 175 pages of documentation showing that they had, in fact, developed a novel way of producing macroscopic amounts of the compounds of the claimed invention.

Over a year later, on September 12, 2008, the Office finally responded with a non-final Office Action drafted by Examiner Chaney. Examiner Chaney allowed claims 89, 91, 120, and 121 and either rejected or objected to the other claims.[6] *See* non-final Office Action, dated September 12, 2008. In objecting to claims 90, 93, 95-97, 99-103, 106, 108, 110-112, 114, 115, 118, Examiner Chaney specifically stated:

> Applicant uses several terms in the claims that **appear to be equivalents at the time of filing** of the instant application. At the time of filing, the term "fullerene" was understood to mean the family of hollow caged carbon molecules with an even number of carbon atoms represented by C_{60}, C_{70}, etc. as stated by Harold Kroto in his declaration filed on 16 November 1999 in the instant application. At the time of filing, the terms "a cage carbon allotrope consisting solely of carbon atoms soluble in non-polar organic solvents," "a cage carbon allotrope consisting solely of carbon atoms soluble in non-polar organic solvents, which allotrope of carbon is neither graphite or diamond," "allotrope of carbon is neither graphite nor diamond," and "a cage carbon

[6] Again, Examiner Chaney indicated claim 120 was allowable, which was directed to "Substantially pure fullerenes in solid form."

> allotrope consisting solely of carbon atoms" can only refer to the carbon allotrope represented by the fullerene family.

(*Id.* at 4) (emphasis added).

On September 23, 2008, in an Examiner interview with Examiners Chaney and Tsang-Foster, Applicants proposed amending the claim language to use the term "cage molecules consisting of carbon atoms soluble in non-polar organic solvents" to address Examiner Chaney's concerns about duplication of subject matter in the claims. *See* Examiner Interview Summary Record, dated September 23, 2008. Examiner Chaney agreed the case could be allowable if the currently pending claims were canceled and claims were drafted mirroring those indicated as allowable in co-pending U.S. Appl. No. 07/580,246. *Id.*.

In response to the Examiner interview, Applicants, in a response filed March 12, 2009, canceled claims 1-121 and added new claims 122-163 which incorporated the claim language "cage molecules consisting of carbon atoms." *See* Amendment, dated March 12, 2009. While this language is not identical to the language proposed in the September 23, 2008 interview, Applicants note that, based on Examiner Chaney's own September 12, 2008 statement, it is equivalent language. Examiner Chaney specifically notes that "[a]t the time of filing, the terms "a cage carbon allotrope consisting solely of carbon atoms soluble in non-polar organic solvents," . . . and "a cage carbon allotrope consisting solely of carbon atoms" can only refer to the carbon allotrope represented by the fullerene family." (*see* page 4 of non-final Office Action, dated September 12, 2008). As such, Applicants believe that the amendment filed March 3, 2009 met the requirements of 37 CFR 1.111(a)(2)(i)(B), because, while it did not use the exact language discussed in the previous interview, it incorporated the changes proposed through equivalent language.

Subsequently, Examiner Hendrickson was (again) re-assigned the case, and in a telephonic interview on March 19, 2009, noted that the pending claims (claims 122-163) would be considered allowable "if Applicants amend the claims to limit the term 'cage' to consist of '60 carbon atoms' or '70 carbon atoms'."[7] *See* Applicant Interview Summary, dated September 25, 2009. However, before Applicants could respond to the substance of the interview with Examiner Hendrickson, the case was reassigned again, this time to Examiner Yuan.

Examiner Yuan chose to completely ignore Examiner Hendrickson's statements regarding allowability of the claims based on incorporation of the limitation of 60 or 70 carbon atoms, instead suggesting the incorporation of "soluble in nonpolar organic solvents" into the pending claims. *See* page 4 of final Office Action. Examiner Yuan stated that he was concerned that without said language, the claims would read on nanotubes and nanowires, which he felt were outside the scope of the present invention. *Id.* at 6. Examiner Yuan offered to draft an amendment with claims containing his preferred claim language and remove what he felt was unsupported language. Applicants agreed to consider the Examiner's amendment.

Applicants received Examiner Yuan's proposed claims on September 11, 2009. After reviewing the claims, Applicants spoke to Examiner Yuan on September 23-24, 2009, proposing acceptance of the Examiner's claims along with a request for consideration of a number of additional claims. Examiner Yuan only grudgingly accepted Applicants' proposal and after reviewing Applicants proposed amendments, was extremely recalcitrant, insisting that Applicants either accept his proposed claims or he would issue a final Office Action. *Id.* Examiner Yuan told Applicants he doubted that support existed for Applicants' proposed language, despite the fact that clear support indeed exists for the proposed language in the

[7] As noted above, Applicants are amenable to this language and indeed it has been incorporated into the claims of

Detailed Description of the application. *See* table of exemplary support in Supplemental Amendment. Applicants could not accept the Examiner's claims without prior approval and requested time to consider the proposed claims.

In response to the Examiner's amendment, and the fact that the Applicants had in no way been able to respond to the previous action on the part of Examiner Hendrickson, Applicants submitted a supplemental response and claim amendments on September 25, 2009. Applicants' response was submitted to account for the unresolved issues stemming from the March 19, 2009 interview with Examiner Hendrickson, to incorporate Examiner Yuan's proposed claims, and to incorporate the alternative claim language which Applicants believe is fully supported by the specification and also addresses Examiner Yuan's concerns about the invention reading on nanotubes.

In what can only be described as an inequitable act given the history of this application, Examiner Yuan refused to enter the Supplemental Amendment. Applicants note that at the time the Supplemental Amendment was submitted, action in this case had been essentially suspended by the Office, and Applicants had dealt with at least three different Examiners and had received conflicting information from all three examiners as to what the Office felt was necessary for allowability of the claims.[8] Further, Applicants were given no opportunity to address the issues raised by Examiner Hendrickson, as should have been allowed to do. Applicants have continually attempted to work in good faith with the Office, and believe that the Examiner's recalcitrance shows a lack of reasonableness in light of the obscure way in which the Office has dealt with this application. Applicants believe that the convoluted and vacillating approach the

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the Supplemental Amendment (see newly proposed claims 203, 204, 216, 217, 224, 225, 236, and 237).

[8] Examiner Chaney indicated allowability for claims to "substantially pure fullerenes"; Examiner Hendrickson indicated allowability for cage moieties "having 60 or 70 carbons"; Examiner Yuan indicates allowability for "cage molecules that are soluble in non-polar solvents."

Office has used in dealing with the subject application is the main reason that the application is still pending and that there is so much confusion and disagreement as to the scope of allowable subject matter. Accordingly, Applicants respectfully request out of equity and fairness that the Office enter Applicants' Supplemental Amendment at this time and permit Applicants an appropriate examination of the proposed claims on the merits.

The Newly Raised §112, First Paragraph Rejection is a Clear Error

As a matter of procedure, Applicants emphasize that this petition merely requests that the Technology Center Director exercise the appropriate authority to facilitate entry and consideration of the Supplemental Amendment filed September 25, 2009, to which Applicants are entitled based on the legal arguments presented above. However, Applicants believe that the Technology Center Director should be aware that the §112, first paragraph rejection newly raised by Examiner Yuan is substantively and legally erroneous.

For example, Examiner Yuan has rejected Applicants' currently pending claims under 35 U.S.C. §112, first paragraph for lack of written description because "the genus of 'cage molecules' also encompasses carbon nanotubes for which there is no written description support in the original disclosure." Final Office Action, p. 12. Federal case law makes it clear, however, that it is impermissible to use a later factual reference to determine whether the application is enabled or described as required under 35 U.S.C. 112, first paragraph. MPEP 2124 (citing *In re Koller*, 613 F.2d 819, 823 n. 5, 204 USPQ 702, 706 n.5 (CCPA 1980)). The present application has a priority date of September 10, 1990. According to published accounts, carbon nanotubes were

first described in November 1991, well over a year after Applicants' invention.[9] It is impermissible – indeed a clear error – for the Office to be reading Applicants' specification in light of post-published art for purposes §112 first paragraph, now matter how tempting that might be given the age of this application.

The Federal Circuit aptly illustrated the error underlying the recently applied §112 first paragraph rejection in *U.S. Steele Corp. v. Phillips Petroleum Co.*, where they stated:

> [D]efendants fail to recognize that "application sufficiency under Sec. 112, first paragraph, must be judged as of the filing date." *In re Glass*, 492 F.2d 1228, 1232, 181 USPQ 31, 34 (CCPA 1974); see *In re Hogan*, 559 F.2d at 604, 194 USPQ at 535 (Sec. 112's enablement requirement); *In re Koller*, 613 F.2d 819, 823, 204 USPQ 702, 706 (CCPA 1977) (Sec. 122's written description requirement).
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> . . .
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> *In re Koller* is particularly illustrative. The claims there at issue and claims contained in a grandparent application contained the broad term "liquid medium." The PTO board held that appellants could not rely on the grandparent's filing date because "[t]he broad recitation 'liquid medium' would have been construed by one skilled in the art from the disclosure as consisting of water or water to which a miscible organic solvent is added." [citation omitted.] Because of a later discovery that water-immiscible solvents could be used, the board emphasized that "[t]he term [liquid medium] as now interpreted by appellants is broader than that disclosed in the grandparent application."
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> . . .
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> Our predecessor court reversed, citing the general rule that "language in a specification is to be understood for what it meant to one having ordinary skill in the art at the time the application was filed," [citation omitted], and noting that support need be found for only the claimed invention, in view of how one skilled in the art at that time would construe the claims and would read its specification.
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> . . .
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> That the '851 claim may cover a later version of the claimed composition . . . relates to infringement, not to patentability. See *In re Hogan*, 559 F.2d at 607, 194 USPQ at 538. To hold differently would, in the words of *Hogan*, "impose an

[9] Ijima, *Nature* **354**, 56–58 (1991).

> impossible burden on inventors and thus on the patent system." 559 F.2d at 606, 194 USPQ at 537.

Applicants see unmistakable parallels between the rejection newly raised by Examiner Yuan and the rejection applied in *In re Koller*. As noted in the final Office Action, "Examiner Yuan told Mr. Barron on September 24, 2009 that proposed claim 164 was not supported by the original disclosure because the *present day meaning* of fullerene encompasses carbon nanotubes which applicants did not invent." Final Office Action, p. 6 (with emphasis). Similarly, as Examiner Yuan states on pages 13-14 of the final Action:

> The species C_{60} and C_{70} and their corresponding higher order family members such as C_{240} are representaive of the genus encompassed by the term fullerene as understood at the time of the effective filing date of the present application by one of ordinary skill in the art. Today, the definition of fullerene has evolved to encompass carbon nanotubes [citation omitted]. Applicants clearly did not conceive of carbon nanotubes (that is, did not have possession of carbon nanotubes) or regard carbon nanotubes as their invention as of the effective filing date or actual filing date of the present application.

Thus, as in *In re Koller*, Examiner Yuan evaluates written description of Applicants' invention in light of a later discovery, not according to the state of the art at the time of filing. In fact, Examiner Yuan acknowledges in the above paragraph that the disclosed species are indeed representative of the claimed genus as viewed as of the effective filing date, the date on which the inquiry is to be based! Indeed, carbon nanotubes have been described as a "by-product" of the discovery of C60 fullerenes, which is a testament to the pioneering nature of Applicants' invention.[10] As the Federal Circuit recognized in *In re Koller*:

> As pioneers, if such they be, they would deserve broad claims to the broad concept. What were once referred to as "basic inventions" have led to "basic patents," which amounted to real incentives, not only to invention and its disclosure, but to its prompt, early disclosure. If later states of the art could be employed as a basis for rejection under 35 U.S.C. § 112, the opportunity for

[10] Abhijit Gokhale, Carbon Nano-Tubes: Properties and Applications, New Jersey Institute of Technology, section 2.I (http://agokhale.com/Documents/Carbon%20Nanotubes.pdf).

> obtaining a basic patent upon early disclosure of pioneer inventions would be abolished. [citing *In re Hogan*, 559 F.2d at 606, 194 USPQ at 537.]
>
> In *Hogan*, an analysis using later-filed references to determine the scope of enablement was found to be impermissible. Similarly, it cannot be allowed when, as here, the description requirement is an issue.

The invention claimed in the present application would certainly be defined as pioneering. Applicants were the first to isolate fullerenes in macroscopic amounts, which opened the way for an entire field of structural chemistry. Thus, while Applicants have endeavored to work with Examiner Yuan on potential claim language to address his concerns about nanotubes, the case law makes it clear that the newly raised §112 first paragraph rejection as applied to the currently pending claims is a clear error.

Summary

In conclusion, Applicants believe that entry of the Supplemental Amendment filed September 25, 2009 is warranted on both legal and equitable grounds. The claims proposed in the amendment both incorporate suggested revisions proposed by Examiner Hendrickson and directly address concerns regarding scope raised by Examiner Yuan. Accordingly, entry of the amendment is warranted under 37 CFR 1.111(a)(2)(B). Moreover, Applicants believe the newly submitted claims more adequately address the §112 issues raised by Examiner Yuan than the claims proposed by the Office and therefore certainly narrow the issues for appeal under 37 CFR §1.111(a)(2)(F).

In any case, entry of the Supplemental Amendment should be made pursuant to 37 CFR §1.112 given that the issues under 35 U.S.C. §112 raised by Examiners Hendrickson and Yuan after the nonfinal Office Action constitute additional examination prior to final to which

Applicants had a right to respond with amendments. Alternatively, the finality of the most recent Office Action dated June 23, 2010, should be withdrawn, given that the "newly raised" rejection under 35 U.S.C. §112, first paragraph, most certainly was not made in view of Applicants' amendment filed March 19, 2009, but rather was a result of an examiner reassignment and a clear departure from prior examination.

Finally, equity warrants entry of Applicants' Supplemental Amendment given the protracted and confusing history of this application, and the vacillating manner in which this application was transferred around to multiple examiners, each agreeing to allow a different scope of claims. It is unreasonable for the Office to now deny Applicants the opportunity to submit additional claims that Applicants believe respond to the issues raised by the various Examiners, for consideration on the merits.

Nevertheless, the newly raised §112 first paragraph rejection entirely contradicts well-established federal case law and should be withdrawn as to the currently pending claims. Given the Examiner's multiple statements on the record regarding the claims reading on structures isolated well after Applicants priority date, it is quite clear that Examiner Yuan is ignoring federal precedent and construing the claim terms not according to what they would have meant to one of ordinary skill in the art at the time the application was filed, but what they might read on in the future.

Applicants respectfully request that the Technology Center Director exercise the appropriate authority to secure entry of the Supplemental Amendment and examination of the newly submitted claims. If at any time Examiner Yuan or the Technology Center Director believes that an additional telephonic interview will help to expedite examination, he or she is encouraged to contact the undersigned.

Except for issue fees payable under 37 C.F.R. 1.18, the Commissioner is hereby authorized by this paper to charge any additional fees during the entire pendency of this application including fees due under 37 C.F.R. 1.16 and 1.17 which may be required, including any required extension of time fees, or credit any overpayment to Deposit Account 50-1283.

Dated: August 23, 2010

Respectfully submitted,
COOLEY LLP

COOLEY LLP
ATTN: Patent Group
777 6th Street NW, Suite 1100
Washington, DC 20001

Tel: (202) 842-7833
Fax: (202) 842-7899

By: Bonnie Weiss McLeod

Bonnie Weiss McLeod
Reg. No. 43,255

New Jersey Institute of Technology
Department of Mechanical Engineering.

Carbon Nano–Tubes: Properties and Applications

Abhijit Gokhale

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1. INTRODUCTION

Natural carbon exists in several forms. Carbon is one of the most abundant elements found in the earth's crust. It can form variety of materials with very different properties. It can appear as a transparent crystal but also as a black amorphous soot its rare to find this diversity. Most know about graphite and diamond, but there is a third type: fullerenes. Since last 15 years intense renewal of interest for carbon science is shown, provoked by the discovery of a hollow cluster of 60 carbon atoms shaped like a soccer ball was discovered in 1985. It is exceedingly rugged and very stable.

The discovery of buckyball in 1985 by Rice University professors Richard Smalley and Robert Curt, and University of Sussex professor Harold Kroto led to a new class of carbon based materials, the fullerenes. Just as diamond and graphite are forms of carbon, so are fullerenes and nanotubes. The buckyball is the most basic fullerene, comprised of 60 carbon atoms arranged in a pattern resembling a soccer ball. As structure is elongated, it forms nanotubes, which is about a nanometer in diameter.

Carbon nanotubes were discovered by Dr. Ijima Sumio of NEC Corp. in 1991 . This is the world's strongest fiber that has been discovered so far. The conductivity of electricity is higher than that of diamond. Also its strength is 100 times greater than steel at one sixth the weight, and high strain to failure. Moreover, it is resistant to extreme heat and is lighter than aluminum. Single – walled carbon nanotubes have extraordinary mechanical, electrical and thermal properties. As individual molecules, single – walled nanotubes are believed to be mostly defect free, leading to high strength, despite their low density

Table for the timeline of carbon nanotubes[C]:

Sr. No	Year	Description
1.	1991	Discovery of multiwall carbon nanotubes
2.	1992	Conductivity of carbon nanotubes
3.	1993	Structural rigidity of carbon nanotubes & Synthesis of single wall nanotubes
4.	1995	Nanotubes as field emitter
5.	1996	Ropes of single wall nanotubes
6.	1997	Quantum conductance of carbon nanotubes
7.	1998	Chemical vapor deposition synthesis of aligned nanotube films & synthesis of nanotube peapods
8.	1999	Hydrogen storage in nanotubes
9.	2000	Nanotubes as ideal thermal conductors & Macroscopically aligned nanotubes
10.	2001	Integration of carbon nanotubes for logic circuits & Intrinsic superconductivity of carbon nanotubes

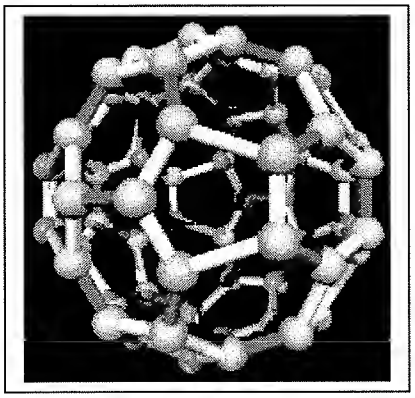
2. C_{60}

The fullerenes are built up of hexagons and pentagons. Only the latter give the curvature to the molecules, actually graphite is flat. The different fullerenes obey to the Euler theorem for polyhedra: f + v = e + 2
where: f is the number of faces, v the number of vertices and e the number of edges . If we have h hexagonal faces and p pentagonal faces we could write :
= p + h
2e = 5p + 6h
3v = 5p + 6h
these three equations lead to p = 12

So for each fullerene with only pentagonal and hexagonal faces there are 12 pentagons and an arbitrary number of hexagons. Thus, the smallest fullerene is C20, built up of 12 pentagons and no hexagon. Nevertheless, when two pentagons are adjacent to each other, there is a high local curvature and so a high strain. Such a situation is not energetically favorable. There is an "isolated pentagon rule "that says that all the pentagons must be separated from each other. The smallest fullerene in which there are not two adjacent pentagons is the C_{60}.

The structure of C_{60} was determined theoretically and experimentally as an icosahedra structure. All C are equivalents. Every pentagon is surrounded by five hexagons (isolated pentagon rule). This is the highest degree of symmetry of any known molecule. In the most stable kekule form:
-the double bonds are located at the junctions of the hexagons
-there are no double bonds in the pentagons
-the bond lengths are 1.4 A for a hexagon edge and 1.46 A for a pentagon edge.

2.1 Difference between C60 and other Fullerene



Investigators after C_{60} soon discovered a whole family of related molecules—clusters as small as C20 and as large as C240. Fullerene cages are about 7-15 angstrom in diameter. Interestingly, each of these structures contains exactly twelve pentagons, while the number of hexagons is arbitrary. The smallest fullerene would be C20, containing twelve pentagons and zero hexagons. But such a structure possesses a great deal of strain. Out of all the fullerenes C60 is the roundest and most symmetrical large molecule known to man. Hence from C60 the elongated nanotubes are formed and not with any other fullerenes.

The structure C60 was determined theoretically and experimentally as an icosahedra structure. Every pentagon is surrounded by five hexagons (isolated pentagon rule). Carbon nano tubes exist as a macro – molecule of carbon, analogous to a sheet of graphite (the pure, brittle form of carbon as in pencil lead). However, when coiled, the carbon arrangement becomes very strong. A C60 or bucky ball is about 7 angstrom in diameter. Carbon nano tubes are a by – product of the discovery of fullerenes. Under a right condition carbon atoms can assemble spontaneously from plasma of an arc discharge and formed closed meshes shaped like footballs and cigars.

Fullerene molecules have a unique structure, while carbon nanotubes may be the strongest material in the world and exhibit significant variations in electronic properties for single walled tubes. Controlled passivation of dangling σ-bands of surface carbon atoms by hydrogen in diamond and diamond-like carbon films results in super-low friction and wear in sliding bearing applications. Moreover, the surfaces of all of these structures exhibit unusual chemical properties, from the negative electron affinity of hydrogen-terminated diamond to the chemical stability of diamond, fullerenes and nanotubes. This morphological flexibility makes carbon-based materials inherently multifunctional. In addition, these materials are compatible with both inorganic and biological systems, making devices based on carbon materials especially attractive.

3. Methods of producing nanotubes

Carbon nanotubes are a by-product of the discovery of fullerenes. Under the right conditions carbon atoms can assemble spontaneously from the plasma of an arc discharge and form closed meshes shaped like footballs and cigars. The beauty of molecules such as carbon-60 which has the same pattern of pentagons and hexagons that is found in soccer balls is in sharp contrast with the atomic chaos from which they form with a surprisingly high yield.

There are three major ways to produce nanotubes:
-carbon arc synthesis
-chemical vapor deposition
-ion bombardment

Carbon nanostructures synthesized with nanocrystalline Ni catalyst from decomposition of methane are investigated by means of transmission electron microscopy (TEM). Two kinds of carbon nanostructures, carbon fibers and bamboo-shaped carbon nanotubes, are observed. The preferential growth direction of grapheme sheets depends on the reaction conditions. The bamboo-shaped carbon nanotubes can be obtained only if the reaction temperature is higher than 1000 K, and carbon fibers can be obtained at lower temperatures.

Chains of C60 molecules are present within single walled carbon nanotubes formed by pulsed laser evaporation of a catalyst-containing graphite target. SWNT's were prepared using a high yield metal catalyzed arc synthesis method. In a typical procedure, a 100 mm long 10 mm diameter graphite rod was machined down to a diameter of 6 mm with a 40 mm deep 3.5 mm diameter hole drilled along the central axis of the thin region. This cavity was packed with a mixture of 4.2 at% Ni and 1.0 at% Y mixed intimately with graphite powder. The composite rod was arc evaporated in a Kratschmer-Huffman soot box. A heat shield (a 200 mm long, 100 mm diameter stainless steel cylinder) was placed around the electrodes to confine the carbon plasma. The evaporation was carried out in a dynamic vacuum with a 660 torr He, with a voltage of 25-30 V and a current of 110-140 A maintained between the electrodes. Nearly all of the SWNT samples that we describe were subjected to subsequent heat treatment during capillary filling experiments.

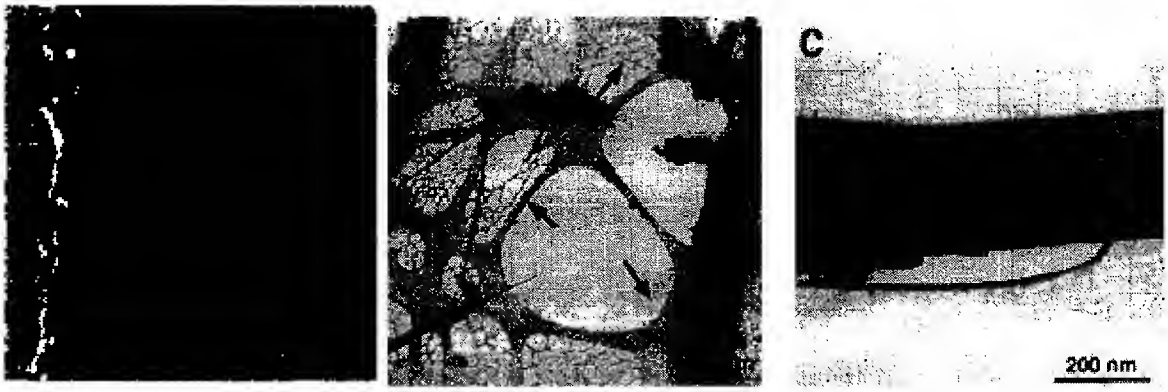
4. Properties of Nanotubes

SWNTs have are stiffer than steel and are resistant to damage from physical forces. Pressing on the tip of the nanotube will cause it to bend without damage to the tip or the whole CNT. When the force is removed, the tip of nanotube will recover to its original state. Quantizing these effects, however, is rather difficult and an exact numerical value cannot be agreed upon.

Common mechanical properties of nanotubes are listed below :

Young's modulus = > 1 TPa (tera Pascal)
Stiffness = same as diamond
Tensile strength = 30 Gpa -----[3]
Tensile strength = 200Gpa -----[4]
E_{gap} = 1.7 to 2eV
Thermal conductivity = 1800-6000 W/m-K (as high as diamond)
Coefficient of conductance, Go = 1/12.9 KOhmE-1
Density = 1.34g/cm2
Lattice parameter = 16.78 Angstroms (armchair), 16.52 Angstroms (zigzag)
Electric conductivity as high as copper
Tensile strength 100 times greater than steel (30 GPa) .

5. Doping of nanotubes



(a) Part of typical nanotubes with B-C-N stoichiometry . (b) Overview of specimen with long flexible tubes which are frequently attached to the holey carbon film holes . (c) Obeservation of highly bend tube .

Carbon nanotubes can be doped either by electron donors or electron acceptors . After reaction with host materials , the dopants are intercalated in the intershell spaces of the multiwalled nanotubes , and in the case of single – wall nanotubes either in between the individual tubes or inside the tubes . The reaction of intercalation can be carried out in the vapor or liquid phase , and electrochemically .

The ternary system B-C-N , has attracted considerable attention recent years due to interesting properties of the phases in this system . The possibility of tailoring the electronic properties of the material is important characteristic of this system . Boron enhances the longitudinal growth of carbon nanotubes increasing both length - to – diameter ratio (by means of a substantial increase of atleast 10 times in length) and the graphitization behavior . Better graphitization due to boron doping leads to high resistance to etching . The tendency to form carbon nanotubes with a higher concentration of B and N is small compared to the yield of boron doped nanotubes .

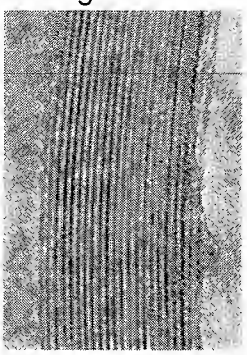
6. APPLICATIONS

Several decades from now we may see integrated circuits with components and wires made from nanotubes, and maybe even buildings that can snap back into shape after an earthquake.
The first commercial device that uses multiwall nanotubes may be a lamp that operates on the field-emission principle.
Research at IBM indicates that nanotubes transistors should be competitive with state-of-the –art silicon devices.
Nanotubes could also be used to store hydrogen to power electric vehicles.

Hydrogen storage in Carbon Nanotubes

Carbon nanotubes technology represents a new direction for solid hydrogen storage as it can be altered to store large amount of hydrogen at room temperature. Carbon nanotubes store hydrogen in microscopic pores on the tubes and within the tube structures. Large diameter single wall nanotubes can be an ideal media for hydrogen storage because of their high gravimetric energy density .

The major advantage of using carbon nanotubes is high surface area carbon structures for absorption. Another advantage of carbon nanotubes is the amount of hydrogen they are able to store. Carbon nanotubes are capable of storing from 4.2% - to 6.5% of their own weight in hydrogen. Thirdly this storage system is reliable, simple to engineer and very safe.



Porous carbons are of interest as molecular sieve materials, both as sorbants and as membranes, or as nanostraws for filtration. One of the major research objectives is to develop materials or structures with exceedingly high storage capacity per unit volume and weight for gases such as H_2 or CH_4.H_2 or CH_4 could become an economic source of combustion fuel or a means to power fuel cells for ultra low-emission vehicles or for electric power generation.

The large diameter single wall nanotubes can be an ideal media for hydrogen storage because of their high gravimetric energy density. Current technology for hydrogen storage methods involve compressing the gas in high-pressure cylinders or the conversion of metals to metal-hydrides, but at the present these procedures are to bulky or too heavy for practical transport applications. Carbon nanotubes may offer a potential solution to this problem as it has been suggested pores of molecular dimensions can adsorb large quantities of gasses owing to the enhanced density of the adsorbed material inside the pores, a consequence of the attractive potential of the pore walls .

Compressed H2 Storage validation tests:

Following are the standard tests taken to approve the hydrogen storage tanks:

- Hydrostatic Burst
- Extreme temperature cycle
- Ambient cycle
- Acid environmental
- Bonfire
- Gunfire penetration
- Flow tolerance
- Accelerated stress
- Drop test
- Permeation
- Hydrogen cycle
- Softening temperature
- Tensile properties
- Resin shear
- Boss end material

CASE STUDY:

The national University of Singapore has published a recent paper in "Hydrogen and fuel cell letter ", New York in Nov. 2001.

	DIAMETER	CAPACITY	TEMPERATURE	PRESSURE
SWNT	1.85 nm	4.2 wt %	Room Temp .	10 Mpa .
Pure SWNT	1.63 to 2 nm.	5 %	133 K	-
Crystalline rope of SWNT	-	H/C ratio 1 to1	80 K (Cryogenic Temp)	> 12 MPa
Li Doped Nad K Doped MWNTs	-	14 %	Room Temp	Room Temp .

FUTURE SCOPE:

The ultimate goal is to develop a low cost hydrogen storage material that has hydrogen capacity more than 6.5 wt %, is stable with hydrogen cycling and possesses favorable thermodynamic and dynamic characteristics suitable for transportation and portable devices application. The US Department of Energy has stated that carbon materials need to have a storage capacity of 6.5% of their own body weight to be practical for transportation uses. Carbon nanotubes and their hydrogen storage capacity are still in the research and development stage. Research on this promising technology has focused on the areas of improving manufacturing techniques and reducing costs as carbon nanotubes move towards commercialization.

Following are the specifications collected by a survey which shows where exactly developments are needed.
Auto maker need: Hydrogen storage solution, Assurance that refueling Infrastructure will be there.
Supplier's need: production volume to reduce costs through economies of scale,
demand sufficient to justify capital expenditures .
Consumer's need: Vehicle that are transparent to own and operate (cost , vehicle range , convenience , refueling ease , reliable) compared to today's Conventional gasoline ICE vehicles , convenient refueling and cost competitive fuel .

COMPARISON OF H2 STORAGE PROPERTIES OF VARIOUS SYSTEMS :

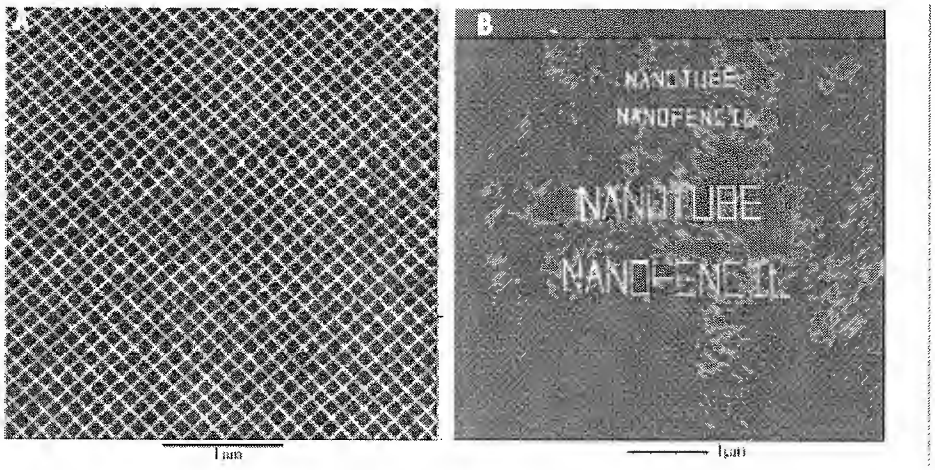
Material	Max wt% H_2	T [K]	p [MPa]
SWNTs (low purity)	5-10	133	0-40
SWNTs (high purity)	8.5	80	7-18
SWNTs (~50 wt% pure)	4.2	300	10-12
GNFs (tubular)	11.26	298	11-35
GNFs (herringbone)	67.55	298	11-35
GNFs (platelet)	53.68	298	11-35
Graphite	4.52	298	11-35
GNFs (herringbone)	0.4	298-773	0-101
Li-GNFs	20.0	473 ~ 673	0-101
Li-Graphite	14.0	473 ~ 673	0101
K-GNFs	14.0	< 313	0-101
K-Graphite	5.0	< 313	0-101
FeTi-H	< 2	> 263	2-5
NiMg-H	< 4	> 523	2-5
Cryoadsorption	~ 5	~ 77	2-5
Isooctane/gasoline	17.3	> 233	0-1

Abhijit Gokhale

FUTURE WORK:

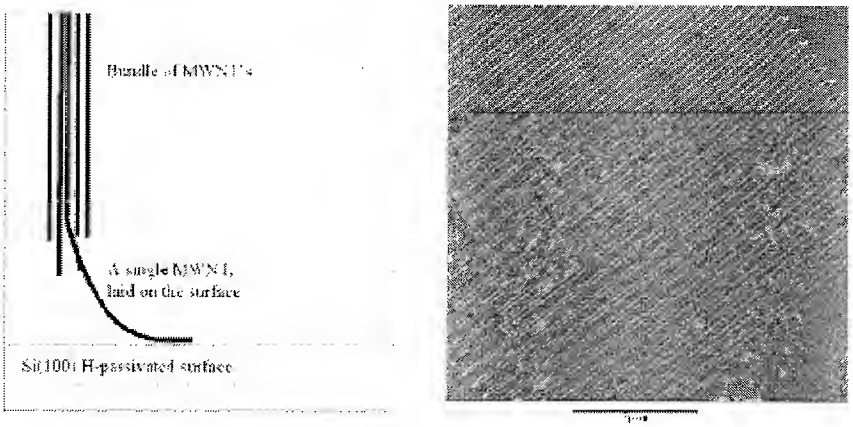
In order to determine the hydrogen sorption performance of these carbon nanotubes preliminary Thermal Desorption Spectrum (TDP) measurements will be performed on samples, using thermo volumetric and thermo gravimetric systems . the rate of hydriding and dehydriding will be obtained at fixed temp and pressures . The stability of the doped SWNT with cycling will be determined. The thermodynamic characteristics of samples will be obtained. The type and size of nanotubes and clusters that result in a reversible high hydrogen capacity will be identified. The effect of contaminants on the performance of hydrogen uptake and release and the capacity of hydrogen will be investigated.

7. Lithography Using Nanotubes :



AFM images of lithography by a nanotube on H – passivated Si (100) surfaces (2 nm tall oxide , 10 nanometer wide lines)
Pattern above written with –9 v tip bias at 10 μm/s scan speed .

High Speed "Nanocalligraphy"



High speed writing possible (upto 500 μm/s) .
Patterns above written with –10 V bias at 200 μm/s oxide height = 0.7ηm , Line width = 10 ηm .
Curved turns due to lateral bending of the tube .

Abhijit Gokhale

8. CONCLUSION

However, many technological hurdles need to be overcome before large-scale applications reach the marketplace. For example, the techniques that are used to build electronic components from nanotubes are painstaking and utterly inappropriate for mass production. But perhaps the most severe limitation is that high-quality nanotubes can only be produced in very limited quantities commercial nanotube soot costs 10 times as much as gold. Although there are many challenges ahead, nanotubes appear destined to open up a host of new practical applications and improve our understanding of basic physics at the nanometer scale. Intense research into carbon nanotubes is sure to continue for at least the next few years.

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